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ION IMPLANTATION IN POLYMERS

by

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*Work supported in part by the Office of Naval Research.

ION IMPLANTATION IN POLYMERS*

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1. Introduction

The purpose of this paper is to introduce the topic of ion implantation in polymers using the perspective of ionic crystals. The scheme of this paper will be a simple introduction of the basic concepts prevalent in polymer studies, followed by a discussion of some of the typical effects of ion implantation and radiation energy deposition in general, with some illustrative examples drawn from recent literature. Many generalizations and a somewhat arbitrary choice of examples are made in the interests of clarity and brevity.

2. Introduction to Polymer Physics:

In general, polymers may be grouped according to their physical properties; thermosetting-, rubber-and thermoplastic-type polymers. Of these, thermoplastic polymers have found the most numerous applications and attracted the most research interest. This type of polymer commonly exists in three phases, listed in order of increasing disorder; glassy, rubbery and viscofluid. The glassy state is characterized by vibrational motion of individual atoms or small segments of the macromolecule. As the temperature of the polymer is increased, the glass transition occurs (Tg) as a distinct endothermic event when intense thermal motion of molecular segments becomes dominant. Increasing temperature can cause a second transition to occur at a point sometimes called the flow temperature, when large scale thermal motion of large parts of the macromolecule becomes characteristic. However, it is not always well defined and in some significant cases - e.g. poly(ethylene oxide), there is merely a monotonic decrease in viscosity as temp increases. An alternative morphology seen most commonly in simple linear molecules is the existence of a crystalline phase, which undergoes melting to the appropriate amorphous state at some well defined melting temperature, Tm.

The crystalline phase has stimulated a lot of interest and it is possible in a few cases, notably polydiacetylene, to produce large single crystals (cm dimensions). This is commonly achieved by solid state polymerization, often using ionizing radiation to affect the polymerization (see, for example Introduction to Polymers" by R. J. Young, [1] Ch. 2.). It is far more common, however, for polycrystalline material to coexist with the appropriate amorphous phase, and the degree of crystallinity of a sample is an important parameter in determining many of the physical properties of these polymers. In such cases, the crystalline regions are often lamellar in structure and the lamellae frequently occur in some form of spherulitic morphology. Since, in a large number of cases, the macromolecules are very "long" in comparison with the thickness of individual lamellae, considerable folding of the molecules occurs. The region surrounding the "folds" in the molecules appears to be more reactive and is involved in the growth of the crystal lawellae under certain circumstances. In addition to these configurational variations, the macromolecular chains have an internal conformation, of which the planar zig zag (e.g. polyethylene) and the helix (e.g. poly(ethylene oxide)) are two which are of particular current interest.

3. Introduction to Implantation Effects.

Having given a brief summary of the structure and morphology to be expected in polymeric materials, it is instructive to consider the effect of the deposition of energy into such systems. Until recently, the damage produced by ion implantation seems to have been the effect of primary interest, however some recent work which also makes use of the doping capabilities of implantation will be mentioned later. The dominant effect of energy deposition is the creation of free radicals, which are, of course, highly reactive. Taking polyethylene as an example, [2], this means that a single neutral hydrogen atom and a free radical carbon are created. Both of these species may be involved in a variety of reac-

tions which typically include crosslinking c_1 scission of the polymer chain, gas evolution and double bond formation e.g. $CH_2 - CH_2 + CH = CH + H_2$.

The evolution of gases of various types, some with relatively complex molecules can be used in various ways. Work by Venkatesan et al [3] suggests a convenient technique for measuring the very low diffusion coefficients (D < 10⁻¹⁰ cm²/s) of some of the larger gas molecules evolved during in ion beam irradiation (e.g. various deuterated formic, acetic and propionic acids in PMMA.) A number of theories have been developed in the chemical literature regarding the exact details of the many possible reactions. When a regard to the probability of crosslinking vs chain scission it has been stated as a general rule that simple unbranched chains will tend to crosslink, whereas similar molecules having large side groups or extensive branching will tend to degrade. As is usually the case, a number of examples contradicting this generalization may easily be quoted.

Changes in susceptibility to oxidation have also been noted in implanted polymers. In considering the polymer during implantation, there is an increased susceptibility to oxidation associated with the presence of sufficient energy in conjunction with the reacting species [4]. Studies of radiation enhanced oxidation have been caried out in, for example, polyethylene [5,6], poly(ethylene oxide) [7] and poly(vinyl chloride) [8,9]. However, once the implantation is complete, there is evidence that some materials, polyacetylene for example, show an enhanced resistance to surface oxidation in the atmosphere

- [10]. In conjunction with the chemical effects, there are a number of physical properties which have been of interest to researchers and a few of those dealt with recently in the literature will serve as examples.
- 4. Applications of Ion Implantation.

In a large number of cases the degree of crystallinity is changed substantially by irradiation. In the case of polymers which form crosslinks under irradiation there is in general an increase in the degree of crystallinity. In the case of polyethylene the crosslinking occurs preferentially in the amorphous phase, however Bhateja et al [11,12] show some rather interesting results of y-irradiating ultra high molecular weight (U.H.M.W.) polyethylene. In the case of such very long molecules, there are significant numbers of tie molecules which tend to inhibit crystallization. y-irradiation preferentially breaks the tie molecules, allowing small scale reorganization among the chains, resulting both in an increase in the perfection of existing crystallites and a growth of additional lamellae. Kusy and Turner [13] report another aspect of crosslinkage formation, in this case in poly(ethylene oxide), namely the depression of the melting point of the crystalline material. The depression, measured by differential thermal analysis, was found to be 0.15 K/Mrad and is attributed to the effective removal of crosslinked units from the equilibrium between crystalline and amorphous material, whose temperature dependence defines the melting temperature.

Another effect attributable largely to the formation of crosslinks is that on the tensile properties of materials. Bhateja and Andrews [14] again working on U.H.M.W. polyethylene noted about a 15% increase in tensile yield stress after 120 MRad of 2 MeV electrons. In addition, the creep strain was observed to be reduced by a factor of five after 64 MRad, which is probably due largely to crosslinking in the amorphous phase, since degree of crystallinity had little effect on this result.

Another physical property which undergoes changes upon irradiation largely because of crosslinking or scission is the solubility, leading to a number of applications in the field of resist materials. In a number of resist materials, both positive and negative resists, the exposure of the resist scales roughly linearly with the linear energy transfer (LET) of the ionizing radiation.

Studies of positive resists such as PMMA [15.7], Komuro et al, MacIver), PMMA plus copolymers like PVA [15], and negative resists such as poly(dimethy/siloxane) [16] and polystrene [15] all show an exhanced exposure rate and excellent resolution [17]. For most positive resists, the exposure mechanism simply involves the scission of the molecular chains resulting in an increased solubility. Of the negative resists, which become less soluble as a result of crosslinking, some actually undergo crosslinking via simultaneous activation of sites on two adjacent chains. In these cases exposure is additionally enhanced by the high energy density surrounding the track of an implanted ion.

Finally, the conducting properties of polymers have become of immense importance as their advantages as solid state electrolytes have become apparent.

Both electron—and ion—conducting polymers have been investigated and studies of ion implantation into electron—conductors have shown an enhancement of conductivity as a result of damage formation and because of reactions with the implanted species.

In the realm of high energy, high dose implantations, the work of Venkatesan et al [18-20] is representative. In this work, doses of 10^{16} - 10^{17} cm⁻² of 2 MeV Ar ions were implanted into PMMA, PVC, a polyimide and some commercial resist materials. Under such conditions there is substantial loss of the target materials, with a decrease to about 50% of the initial film thickness. At doses between 10^{14} and 5×10^{15} cm⁻² the conductivity of the polymers is shown to increase approximately linearly with dose, over 12 orders of magnitude and saturate at a dose of about 10^{16} cm⁻². This behavior appears to be a general characteristic of a large number of polymer and "organic" materials [21,22]. After implantation of 10^{16} - 10^{17} cm⁻² Ar ions, Raman spectra indicate that the material is highly disordered with evidence of the existence of crystallites similar to amorphous carbon. The temperature dependence of the conductivity seems to follow an $\exp[T^{-1/2}]$ function.

rather that the exp $[T^{-1/4}]$ seen in amorphous semiconductors and comparison is drawn with the work by Sheng & Abeles [23,24] on hopping conduction in metal grains dispersed in an insulating medium. It appears, therefore that at such high doses and energies the effects are largely due to damage effects in graphitized polymers, whose original structure is largely irrelevant.

Similar results have been seen by Mazurek et al [25,26] in poly(p-phenylene sulphide) bombarded with 100 KeV 75 As and 815 Kr. However, they also include a careful characterization of the samples after bombardment as well as the results of chemical doping implantations using 100 KeV 80 Br ... Poly(p-phenylene sulphide) doped with AsF5 is known to have useful electron-conducting properties, in addition to some processing advantages, despite the fact that the conductivity is unstable in a moist atmosphere. Chemical doping with bromine is also known to enhance the electron conducting properties of a number of polymers and the purpose of the experiments was to see if similar chemical effects could be produced by ion implantation. Instead of the more conventional four probe conductivity measurements, which can be unreliable when applied to thin films, the more elegant technique of spin casting the polymer film over a planar interdigitated electrode structure was used. Current vs voltage curves for the As and Br implantations show ohmic behavior up to about 5 volts, above which space charge effects are observed. Again, the conductivity increases up to about 1015 No appreciable difference is observed between the Kr, producing damage only, and the As which might be supposed in interact chemically with the polymer. However, the implanted films did shown an enormously improved resistance to degradation in air. Perhaps surprisingly, the Br implanted samples do indeed show evidence of chemical activity, having consistently higher conductivities after doses of 10^{16} cm⁻² [Fig. 1] The temperature dependence of the conductivity for both Kr and Br implanted samples show interesting behavior,

having a discontinuity at 150K [Fig. 2]. Such behavior has not been observed in any chemically doped samples. Both sections of the Kr curve and the lower temperature portion of the Br curve indicate a thermally activated conduction mechanism, however the high temperature portion of the Br curve is fitted to a $ln\sigma = BT^{-m}$ giving a value of m in the range 0.2-0.6. This latte may indicate that a variable range hopping mechanism becomes dominant about studies of the As and Kr implanted samples indicated extensive < slinking in the polymer but little chemical bonding of the implanted specie. le Auger spectroscopy indicates some sulfur depletion in the face layers. In this work, the possibility of the enhanced conductivity being due to the carbon rich surface layer was eliminated by removal of that layer and a subsequent measurement of the same bulk conductivity. Other work by this group [26] using other halogen ions, shows a consistent enhancement of electronic conductivity, which appears to scale approximately with the electronegativity of the species, once the damage induced conductivity effect has reached saturation. In all cases the discontinuity in σ as a function of temperature occured at 150K suggesting some type of implantation induced phase transition.

The work by Weber et al [10,29-29] concentrates on the chemical doping effects of ion implantation, thus far dealing with a number of halogen ions and various "inert" ions implanted into polyacetylene at energies from 10-40 KeV and doses up to 10¹⁸ cm². XPS (ESCA) investigations show that the halogens occupy a single type of site, bonded to the polymer backbone. NMR studies of ¹⁹F implanted polyacetylene [29] indicate that esentially all the implanted ions remain within the target and that the chlorine nuclei are well dispersed through the polymer. It is again noted that the normally rather unstable polyacetylene samples showed a marked resistance to decomposition in air. Similar studies of fluorine and carbon tetrafluoride (1 KeV) implanted into polyacetylene, polybuta-

diene and polystyrene by Rabalais et al [30] using XPS also show that the fluorine ions bond chemically to the polymer backbone. Both -CHF- and -CF₂-environments are produced, regardless of the precise form of the bombarding ion or the exact nature of the target polymer. Again, the implanted films were resistant to degradation in air, and indeed, a small XPS peak from oxygen contamination was almost eliminated during irradiation.

In summary, then, there are a remarkably large variety of phenomena related to ion implantation into polymers. The convenience and processability of polymers coupled with the enormous variety of materials ilable, have already lead to extensive use in almost all industrial fields. Probable applications of implanation techniques are already apparent in for example, mask technology and development of polymer electrolytes. The effects both of damage formation and of chemical modification are sources of intensely interesting work and highly promising applied research.

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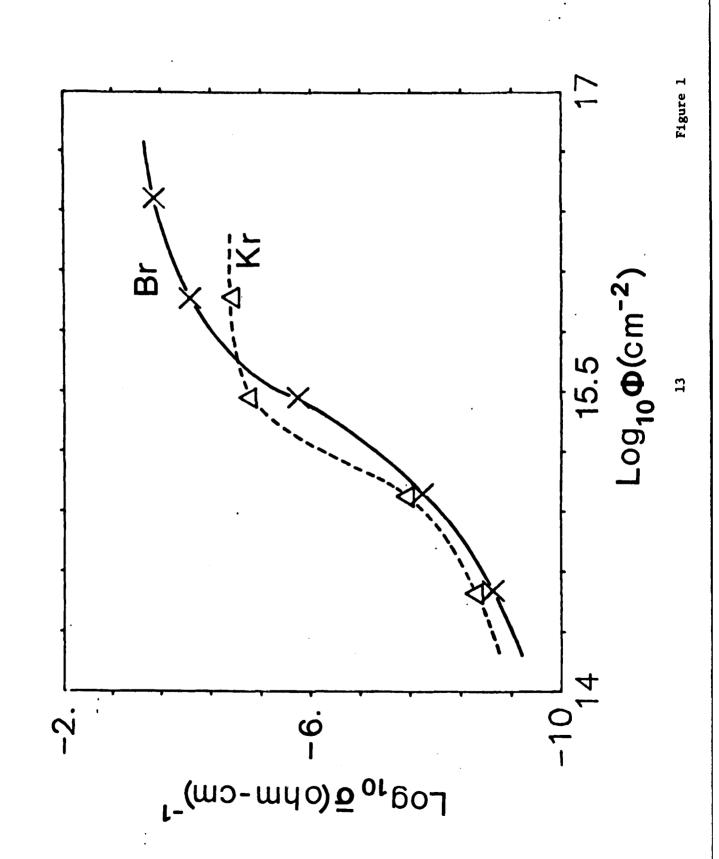
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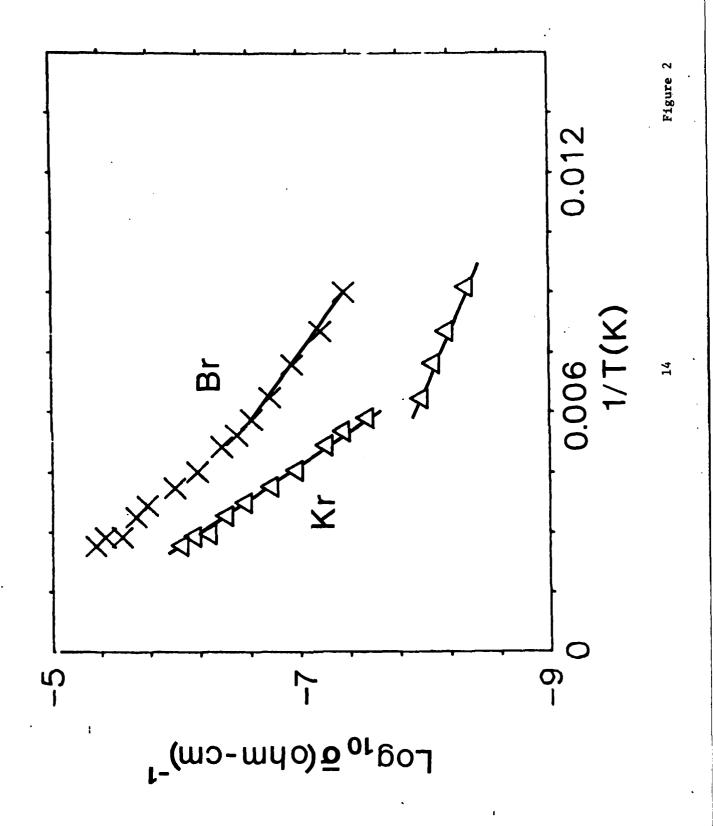
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Figure 1. Log-log plot of mean conductivity-fluence characteristics of $0.2-\mu m$ thick PPS films implanted with bromine ions. The mean conductivity-fluence data for krypton-implanted PPS films are shown for comparison. (Ref. 25)

Figure 2. Log of the mean conductivity vs. reciprocal temperature for bromine-and krypton-implanted PPS films. These data have been interpreted in terms of a $\sigma \sim \exp(-T^{-m})$ functional form. The discontinuity of each curve suggests a phase transition in the PPS host and corresponds to a temperature of about 150 K. (Ref. 25)

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